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Structure of melt-spun Al-2.5Ti-2.5Fe-2.5V alloy and structural changes during annealing①

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Abstract: Rapidly solidified Al-2.5Ti-2.5Fe-2.5V alloy was prepared by melt spinning. As-spun and as-annealed microstructures were studied by TEM, HREM, XRD and EDS analysis. The microhardness of the alloy at different annealing temperature was measured. The results show that the as-spun microstructure of the alloy includes four kinds of primary phases: $Al_{11}(V, Ti)$, $AlTi_3$, $Al_{80}(V, Ti)_{20}$ and $Al_{82}(Fe, Ti, V)_{18}$ phase. After annealing at 300 ℃ for 10 h, metastable phase $Al_{82}(Fe, Ti, V)_{18}$ transforms to the stable phase $Al_{13}Fe_4$. After annealing at 400 ℃ for 10 h, the primary phases $Al_{11}(V, Ti)$ and $AlTi_3$ has completely transformed to $Al_{80}(V, Ti)_{20}$ and $Al_{23}Ti_9$ phase, respectively. The addition of element V increases the microhardness of melt-spun Al-2.5Ti-2.5Fe alloy at elevated temperatures.

Key words: melt spinning; Al-Ti-Fe-V alloy; microstructure; structural change

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1 INTRODUCTION

Rapidly solidified Al-Ti system is one of the novel elevated temperature aluminium alloys with high strength and reduced density[1－10]. As for rapidly solidified Al-Ti system, much attention was paid to the Al-Ti-Fe alloys. In recent years, Kawamura et al[5－7] found that the addition of the transition metal V into the gas atomized Al-Ti-Fe alloy (cooling rate $10^2－10^3$ K/s) can improve its thermal stability and elevated-temperature strength. However, the study on the microstructures of the rapidly solidified Al-Ti-Fe-V alloy and the structural changes during the annealing has not been reported yet. It is known that the increase of the cooling rate can cause the modifications of the rapidly solidified microstructures. The objective of this study is to investigate the microstructures of melt-spun Al-2.5Ti-2.5Fe-2.5V alloy (cooling rate $10^5 \sim 10^6$ K/s), and make clear the structural changes during the annealing.

2 EXPERIMENTAL

Alloy with a nominal composition of 2.5% Ti, 2.5% Fe, 2.5% V (mole fraction, %) and balance Al, was prepared from Al-10Ti alloy (mass fraction, %), V (purity of 99.99%), Fe (purity of 99.9%) and Al (purity of 99.9%) by induction melting under a vacuum atmosphere. Melt-spun ribbons of the alloy were produced by high frequency induction melting under the inert atmosphere of argon. The melt was ejected onto the surface of a rotating copper wheel and the surface velocity was 50 m/s. The width of the prepared ribbons was approximately 3－4 mm, and the thickness 20－40 μm. Several sections of the ribbon were selected and put into the quartz tubes, and the tubes were evacuated to be in vacuum, puffed with the inert argon and sealed. The ribbons in the tubes were annealed at 300 ℃, 400 ℃, 450 ℃ and 500 ℃ for 10 h, respectively. The structures of as-quenched and as-annealed alloys were analyzed by X-ray diffractometry (XRD), transmission electron microscopy (TEM) and high resolution transmission electron microscopy (HREM). Analysis of the chemical composition was carried out using energy-dispersive X-ray spectroscopy (EDS). Thin foil specimens for transmission electron microscopy were prepared from discs 3 mm in diameter cut from the ribbons. The discs were electropolished in a 10% perchloric acid-20% glycerol-70% ethyl alcohol solution. A 71 Vickers hardness tester was used to measure the ambient-temperature microhardness of the alloys annealed at different temperatures. The measured region was near the chill surface of the ribbon. The load was 50 g and the time of loading was 15 s.

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3 RESULTS AND DISCUSSION

3.1 Phases of as-spun and as-annealed Al-2. 5Ti-2. 5Fe-2. 5V alloy

Fig. 1 shows the XRD patterns of the as-spun and as-annealed Al-2. 5Ti-2. 5Fe-2. 5V alloy. XRD analysis reveals that the as-spun alloy predominantly contains α(Al) and four kinds of primary phases: $Al_{11}V$, $AlTi_3$, $Al_{80}V_{20}$ and $Al_{82}Fe_{18}$. The primary phase $AlTi_3$ and $Al_{82}Fe_{18}$ reflections are relatively weak, indicating that the amount of Ti and Fe soluble in aluminum are high, and the amount of precipitations are less. After annealing at 300 ℃ for 10 h, the primary phase $Al_{11}V$ reflections are weakened. The $Al_{80}V_{20}$ reflections are strengthened, and $Al_{23}Ti_9$ reflections appear, while the primary phase $AlTi_3$ reflections cannot be identified. The phenomena mean that the primary phase $Al_{11}V$ begins to transform into $Al_{80}V_{20}$, and $AlTi_3$ transforms into $Al_{23}Ti_9$ completely. Moreover, the $Al_{82}Fe_{18}$ reflections are weakened, and $Al_{13}Fe_4$ appears in the structure, which implies that metastable phase $Al_{82}Fe_{18}$ begins to transform into stable phase $Al_{13}Fe_4$. After annealing at 400 ℃ for 10 h, $Al_{11}V$ reflections disappear, and $Al_{80}V_{20}$ and $Al_{23}Ti_9$ reflections are strengthened further, which means that the primary phase $Al_{11}V$ transforms into $Al_{80}V_{20}$ completely, and Ti element soluble in aluminum keeps precipitating in the form of $Al_{23}Ti_9$. In addition, the $Al_{82}Fe_{18}$ reflections cannot be identified, which indicates that metastable phase $Al_{82}Fe_{18}$ transforms into stable phase $Al_{13}Fe_4$ entirely. After annealing at 450 ℃ and 500 ℃ for 10 h, the $Al_{23}Ti_9$, $Al_{80}V_{20}$ and $Al_{13}Fe_4$ reflections become strengthened further. This may be due to the coarsening of $Al_{23}Ti_9$, $Al_{80}V_{20}$ and $Al_{13}Fe_4$.

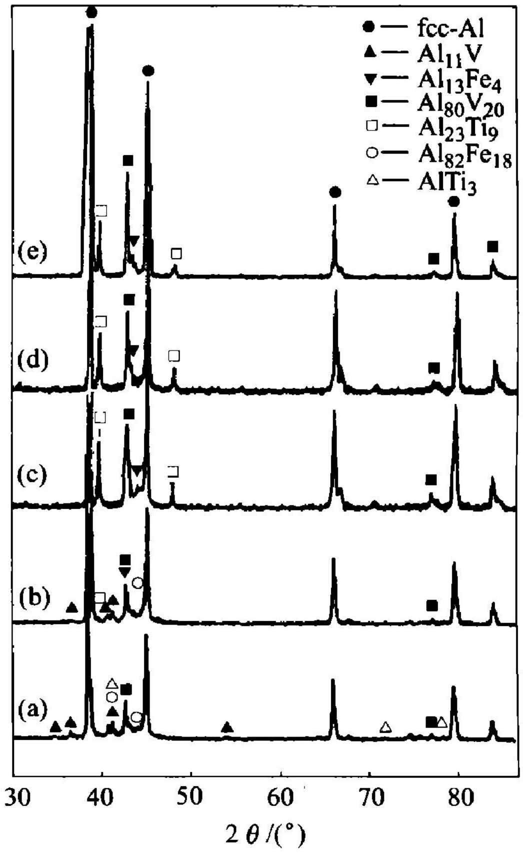

Fig. 1 XRD patterns of as-spun and as-annealed rapidly solidified Al-2. 5Ti-2. 5Fe-2. 5V alloy
(a) —As-spun; (b) —300 ℃, 10 h; (c) —400 ℃, 10 h; (d) —450 ℃, 10 h; (e) —500 ℃, 10 h;

3.2 Microstructures of as-spun Al-2. 5Ti-2. 5Fe-2. 5V alloy

Fig. 2 shows the transmission electron micrograph of the as-spun Al-2. 5Ti-2. 5Fe-2. 5V alloy. It can be seen that spherical intermetallic particles with a size ranging from 150 to 200 nm, and fine dispersion particles with a size about 30 nm are distributed in α(Al) matrix. Clearly, there are two forms of fine dispersion particles: one is little spherical particle, and the other is sticklike particle. Fig. 3(a) shows the SAED pattern of little spherical particle(arrow in the Fig. 2) in the structure, which is identified to be $Al_{11}V$. EDS and HREM analysis (Fig. 3(b), (c)) confirms further that the little spherical particle is presumably $Al_{11}(V, Ti)$. The EDS analysis (Fig. 4(a)) of sticklike particle shows that the phase is composed of Al, Fe, Ti, and V. The composition of

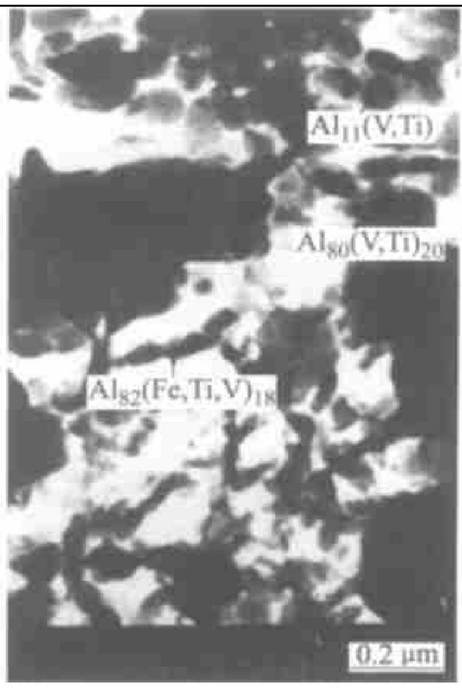

Fig. 2 TEM image of as-spun Al-2. 5Ti-2. 5 Fe-2. 5V alloy

them is determined to be 82.96%Al, 15.47%Fe, 0.51%Ti and 1.07% V (mole fraction). Combining with the X-ray diffraction results, we assume that the sticklike particle is $Al_{82}(Fe, Ti, V)_{18}$. The EDS analysis (Fig. 4(b)) of the spherical intermetallic particle with a size about 200 nm shows that the phase is

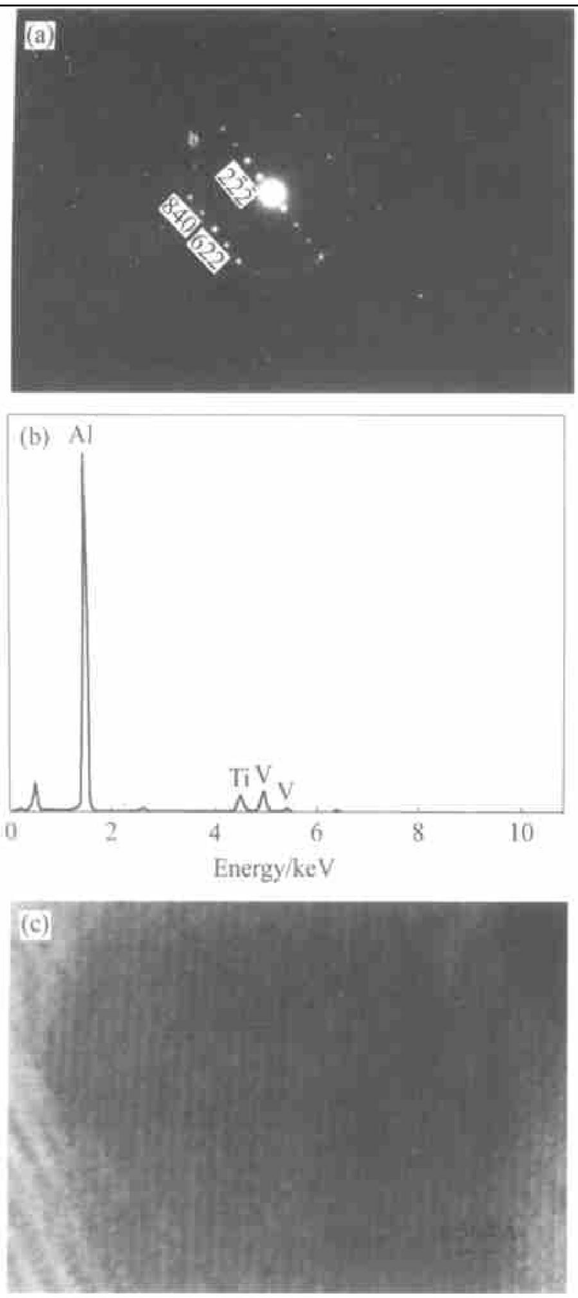

Fig. 3 Characterization of $Al_{11}(V, Ti)$ in as-spun Al-2.5Ti-2.5Fe-2.5V alloy
(a) —SAED; (b) —EDS; (c) —HREM

composed of Al, V and Ti. The composition of them is determined to be 91.35% Al, 4.56% V and 4.09% Ti (mole fraction). Combining with the X-ray diffraction results, we assume that the particle is $Al_{80}(V, Ti)_{20}$.

3.3 Microstructures of as-annealed Al-2.5Ti-2.5Fe-2.5V alloy

Fig. 5 shows the transmission electron micrographs of the as-annealed rapidly solidified Al-2.5Ti-2.5Fe-2.5V alloy at different annealing temperatures. After annealing at 300 ℃ for 10 h, the microstructure of the alloy shows no obvious change in comparison with that of the as-spun alloy. After annealing at 400 ℃ for 10 h, the little sticklike phase with a size about 0.5 μm, identified as $Al_{13}Fe_4$ (Fig. 5

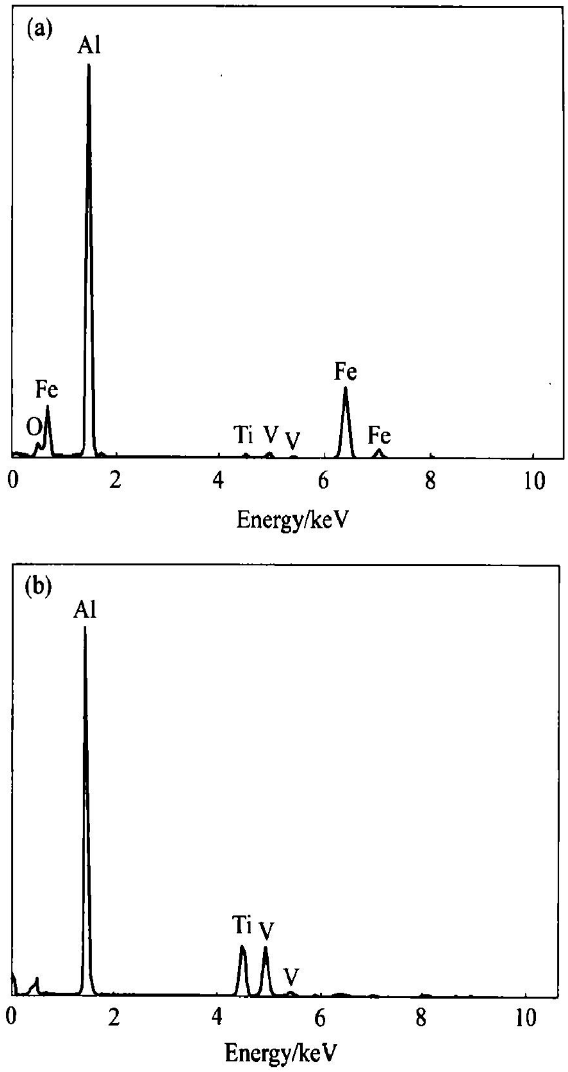

Fig. 4 EDS analysis of phases in as-spun Al-2.5Ti-2.5Fe-2.5V alloy
(a) —$Al_{82}(Fe, Ti, V)_{18}$; (b) —$Al_{80}(V, Ti)_{20}$

(e)), and the spherical particles with a size about 0.1 μm, identified as $Al_{23}Ti_9$ (Fig. 5(f)), appear in the structure (arrow in Fig. 5(b)). This suggests that the transformation of metastable phases into stable phases occurs due to thermal activation. After annealing at 450 ℃ for 10 h, the little sticklike phase $Al_{13}Fe_4$ and the spherical particles $Al_{23}Ti_9$ coarsens further (arrow in Fig. 5(c)). After annealing at 500 ℃ for 10 h, the sticklike $Al_{13}Fe_4$ and the spherical $Al_{23}Ti_9$ particles become coarser greatly. Moreover, it can be seen that the coarsening rate of sticklike phase is larger than that of spherical phase.

It is well recognized that high diffusivity of the solute in alloy matrix and high dispersoid/matrix interfacial energy are two main factors resulting in the coarsening of the dispersoids [11, 12]. The diffusivity of the solute increases with the increase of temperature. Therefore, the higher the annealing temperature, the more severe the coarsening of the dispersoid is. The coarsening rate of sticklike phase ($Al_{13}Fe_4$) may be attributed to the larger

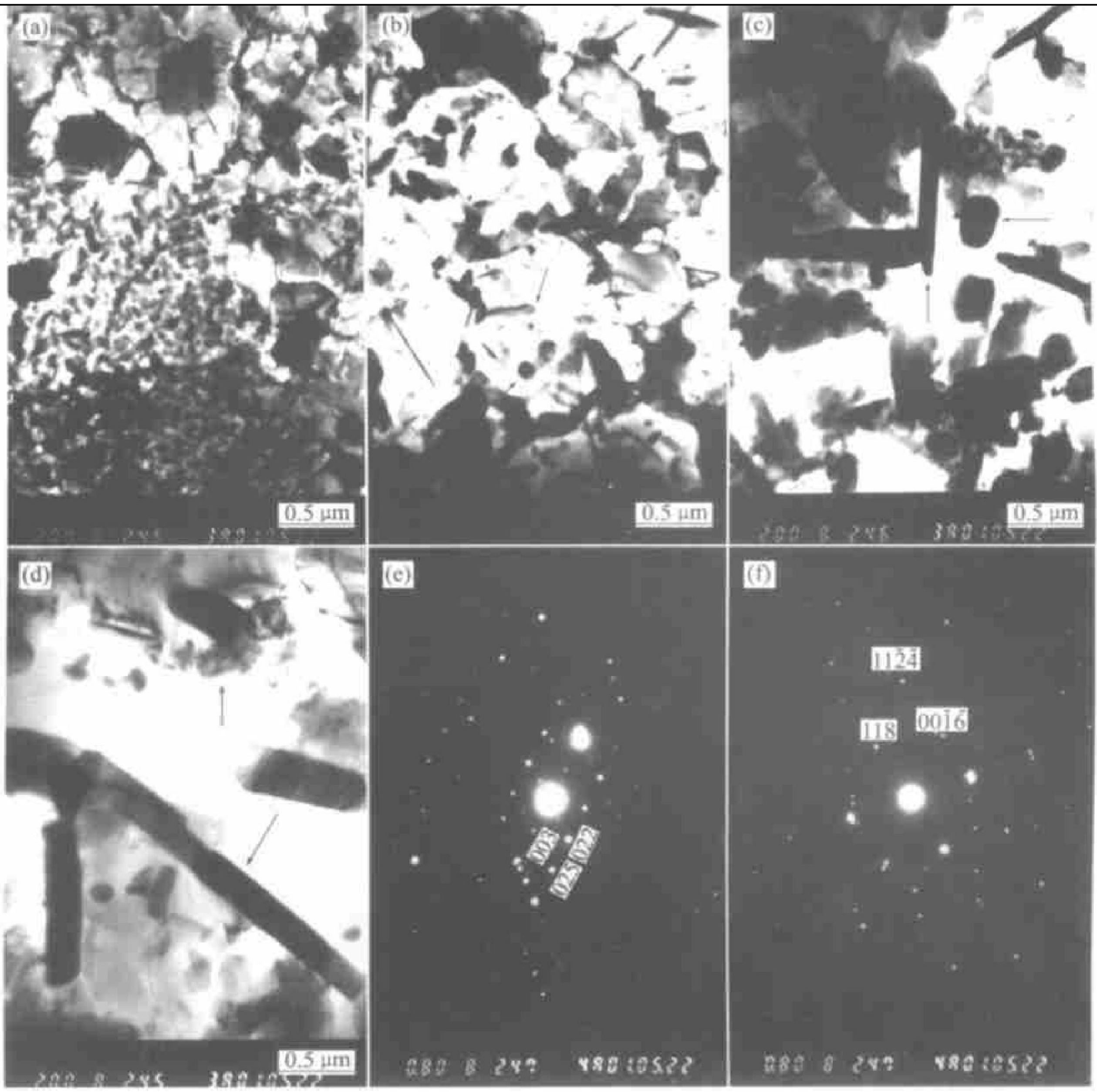

Fig. 5 TEM images and SAED patterns of as-annealed Al-2.5Ti-2.5Fe-2.5V alloy
(a) —300 ℃, 10 h; (b) —400 ℃, 10 h; (c) —450 ℃, 10h; (d) —500 ℃, 10 h;
(e) —Diffraction pattern for $Al_{13}Fe_4$; (f) —Diffraction pattern for $Al_{23}Ti_9$

interfacial energy between the $Al_{13}Fe_4$ and matrix (α(Al)).

3.4 Microhardness of rapidly solidified Al-2.5Ti-2.5Fe-2.5V alloy

Fig. 6 shows the dependence of the microhardness of rapidly solidified Al-2.5Ti-2.5Fe-2.5V alloy on annealing temperature (annealing time is 10 h), here rapidly solidified Al-2.5Ti-2.5Fe as reference material. Clearly, the addition of V element is beneficial to the increase of microhardness of the Al-2.5Ti-2.5Fe alloy at elevated temperature. Because of the addition of V element, the primary phase Al_{11}(V,Ti) and $AlTi_3$, dispersive and fine, are formed in the α(Al) matrix. On the other hand, the addition of V element also prevents the sticklike phase Al_5Ti_2 (with a size about 0.4 μm) from appearing in the

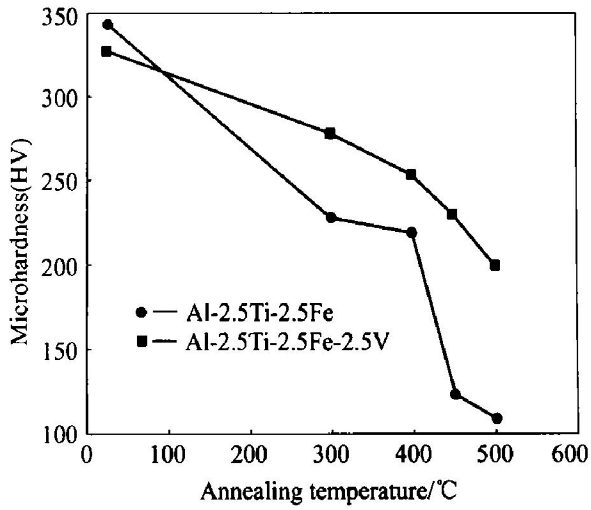

Fig. 6 Dependence of microhardness on annealing temperatures in rapidly solidified Al-2.5Ti-2.5Fe-2.5V alloy

structure of the alloy [6, 13]. Moreover, it can be seen that the microhardness of rapidly solidified Al-2.5Ti-2.5Fe-2.5V alloy decreases slightly with the increase of annealing temperature. On the one hand, this is attributed to the instability of Al_{11}(V, Ti), $AlTi_3$ and Al_{82}(Fe, Ti, V)$_{18}$. When the annealing temperature is higher than 300 ℃, Al_{11}(V, Ti) begins to transform into Al_{80}(V, Ti)$_{20}$, $AlTi_3$ into $Al_{23}Ti_9$, and Al_{82}(Fe, Ti, V)$_{18}$ into $Al_{13}Fe_4$. On the other hand, the coarsening of these dispersoids is also responsible for the decrease of the microhardness at elevated temperature.

4 CONCLUSIONS

1) There are α(Al) and four kinds of primary phases, Al_{11}(V, Ti), $AlTi_3$, Al_{80}(V, Ti)$_{20}$ and Al_{82}(Fe, Ti, V)$_{18}$ in the as-spun microstructure of the melt-spun Al-2.5Ti-2.5Fe-2.5V alloy.

2) There is no obvious change in the structure of the rapidly solidified Al-2.5Ti-2.5Fe-2.5V alloy annealed at the temperature less than 300 ℃. After annealing at 300 ℃ for 10 h, the metastable phase Al_{82}(Fe, Ti, V)$_{18}$ begins to transform into stable phase $Al_{13}Fe_4$. After annealing at 400 ℃ for 10 h, the primary phases Al_{11}(V, Ti) and $AlTi_3$ completely transforms into Al_{80}(V, Ti)$_{20}$ and $Al_{23}Ti_9$, respectively. After annealing at 450 ℃ and 500 ℃ for 10 h, the $Al_{23}Ti_9$, Al_{80}(V, Ti)$_{20}$ and $Al_{13}Fe_4$ become coarser further.

3) The addition of V is beneficial to the microhardness of the rapidly solidified Al-2.5Ti-2.5Fe alloy, especially at high temperature.

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